

Claims:

1. A process for preparing a catalyst active for the fluid bed acetoxylation of ethylene to produce vinyl acetate, which process comprises the steps of :
 - (a) impregnating microspheroidal silica support particles by the incipient wetness technique with an aqueous solution of palladium and gold compounds, whilst
5 agitating the support particles;
 - (b) drying the impregnated support particles produced in step (a) whilst agitating the impregnated support particles;
 - (c) reducing the palladium and gold compounds of the impregnated support particles produced in step (b) to respective metals by adding the dried,
10 impregnated support particles to an aqueous solution of hydrazine, whilst stirring, to form a slurry;
 - (d) filtration of the slurry produced in step (c) to remove the excess reduction solution;
 - (e) washing the filter cake/ slurry produced in step (d) with water and removing
15 excess water to form a cake;
 - (f) impregnating the cake produced in step (e) with one or more salts of Group I, Group II, lanthanide and transition metals by blending the cake produced in step (e) with one or more solid salts of Group I, Group II, lanthanide and transition metals; and
 - 20 (g) drying the impregnated cake produced in step (f) whilst agitating the impregnated cake to form free-flowing catalyst particles.
2. A process according to claim 1, wherein in step (a) the microspheroidal silica

support particles are impregnated by the incipient wetness technique whilst continuously agitating the support particles.

3. A process according to claim 1 or claim 2 in which the palladium compound is selected from the group consisting of palladium acetate, sulphate, nitrate, chloride,
5 halogen-containing palladium compounds and Group I and II salts of halogen-containing palladium compounds.

4. A process according to any one of claims 1 to 3 wherein the gold compound is selected from the group consisting of gold chloride, dimethyl gold acetate, barium acetoaurate, gold acetate, tetrachloroauric acid and Group I and II salts of
10 tetrachloroauric acid.

5. A process according to any one of claims 1 to 4 in which step (b) comprises agitating the impregnated support particles whilst applying external heat at a temperature in the range 50 to 200°C.

6. A process according to any one of claims 1 to 5 wherein in step (g) the cake is
15 dried at a temperature in the range from 60 to 150°C.

7. A process according to any one of the preceding claim in which the microspheroidal support particles are selected from the group consisting of silica, alumina, zirconia and mixtures thereof.

8. A process as according to any one of the preceding claims in which step (a) and
20 step (b) are performed in the same apparatus, said apparatus comprising a vessel being capable of being heated and agitated simultaneously.

9. A process according to claim 8 wherein step (a) and step (b) are performed in an agitated blender.

10. A process according to any one of the preceding claims in which step (f) and
25 step (g) are performed in the same apparatus, said apparatus comprising a blender.

11. A process according to any one of the preceding claims wherein the concentration of hydrazine in the aqueous solution is 1 to 20 wt%.

12. A process according to claim 11 wherein the concentration of hydrazine is 3 to 20 wt%.

30 13. A process according to any one of the preceding claims wherein the aqueous solution of hydrazine has not been rendered alkaline by an alkali metal hydroxide.

14. A process according to any one of the preceding claims comprising preparing a

catalyst containing at least about 0.1wt% to about 5wt% palladium and about 0.1 to about 3wt% gold. EPO - DG 1

15. A process for impregnating microspheroidal catalyst support particles with at least one compound of a catalytically active group VIII noble metal, which process

11.05.2004

94

5 comprises the steps of:

(a') impregnating the microspheroidal support particles by the incipient wetness technique with an aqueous solution of the at least one catalytically active group VIII noble metal, whilst agitating the support particles; and

10 (b') drying the impregnated support particles produced in step (a') whilst agitating the impregnated support particles;

wherein steps (a') and (b') are performed in the same apparatus, said apparatus comprising an agitated blender.

16. A process according to claim 15 in which step (b') comprises agitating the impregnated support particles whilst applying external heat at a temperature in the range 15 50 to 200°C.

17. A process according to claim 15 or claim 16 in which the microspheroidal support particles are selected from the group consisting of silica, alumina, zirconia and mixtures thereof.

18. ~~A process according to any one of claims 15 to 17 in which step (a') and step (b') are performed in the same apparatus, said apparatus comprising an agitated blender.~~

19. ~~18.~~ A process according to any one of claims 15 to ~~18~~17 in which the at least one catalytically active group VIII noble metal comprises palladium.

20. ~~19.~~ A process according to any one of claims 15 to ~~19~~18, further comprising a step (c'), which comprises, contacting the impregnated support particles with a reducing agent to convert the at least one compound to its respective metal.

21. ~~20.~~ A process according to claim ~~20~~19 wherein the impregnated support particles are added to a solution of a reducing agent active for reduction of the at least one metal compound to its respective metal whilst stirring.

22. ~~21.~~ A process according to claim ~~20~~19 or claim ~~21~~20 wherein the reducing agent comprises an aqueous solution of hydrazine.

23. ~~22.~~ A process according to claim ~~22~~21 wherein the concentration of hydrazine in the aqueous solution is 1 to 20 wt%.

- 24-23. A process according to claim 2322 wherein the concentration of hydrazine is 3 to 20 wt%.
- 25-24. A process according to any one of claims 2221 to 2423 in which the at least one compound of a catalytically active metal impregnated in the support comprises
5 palladium and gold compounds.
- 26-25. A process according to any one of claims 1 to 14 and 12 to 2524 in which unreacted hydrazine washed from the material after the reduction step is decomposed in the absence of an oxidant over a supported ruthenium catalyst to nitrogen and ammonia.
- 27-26. A process according to any one of claims 20-19 to 2625, further comprising a
10 step (d') wherein the support particles are further impregnated with one or more salts of Group I, Group II, lanthanide and transition metals, by blending the particles with one or more solid salts of Group I, Group II, lanthanide and transition metals in the presence of a solvent for the salt in which the solvent is contained within the pore volume of the catalyst support particle.
- 15 28-27. A process according to claim 2726 wherein the solvent is water.
- 29-28. A process according to claim 2726 or claim 2827 which further comprises a step (e') wherein the impregnated particles are dried at a temperature in the range from 60°C to 150°C.
- 30-29. A process for the purification of a waste stream comprising dilute aqueous
20 hydrazine, which process comprises contacting the waste stream with a catalyst active for the decomposition of the hydrazine to nitrogen and ammonia; said catalyst comprising ruthenium on a support.
- 31-30. A process according to claim 3029 in which the amount of ruthenium on the support is in the range from 1 to 10 % by weight.
- 25 32-31. A process according to claim 3029 or claim 3130, wherein the support is an inorganic oxide, activated carbon or graphite.
- 33-32. A process according to claim 3231 wherein the inorganic oxide is selected from the group consisting of silica, alumina, zirconia and mixtures thereof.
- 34-33. A process for impregnating porous microspheroidal particles with one or more
30 salts of Group I, Group II, lanthanide and transition metals which process comprises blending the particles with one or more solid salts of Group I, Group II, lanthanide and transition metals in the presence of a solvent for the salt in which the solvent is contained within the pore volume of the support particle.

~~35.34.~~ A process according to claim 3433 wherein the solvent is water.

~~36.35.~~ A process according to claim 3433 or claim 3534, which further comprises the step of drying the impregnated particles at a temperature in the range from 60°C to 150°C.

5 ~~37.36.~~ A process according to any one of claims 3433 to 3635 wherein the blending is performed in a blender.

~~38.37.~~ A process according to any one of claims 3433 to 3736 in which the microspheroidal support particles are selected from the group consisting of silica, alumina, zirconia and mixtures thereof.

10

15

20

25

30